

Chemex

CONTRACT REPORT NO. OSR84-00205

REQUISITION NO. 23241-4-1697

TITLE: "ANALYSIS OF FISH MEAL FOR
RADIONUCLIDES - I

CONTRACTOR: CHEMEX LABS. LTD.

TABLE OF RESULTS

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FISH MEAL

Isotope	Activity (Bq/g)	Uncertainty (Bq/g, 95%C.L.)	Subsample Mass(g)	Date of Analysis
Pb - 210	0.013	0.007	5.000	Mar. 18, 1985
Pb - 210	0.012	0.007	5.000	Mar. 18, 1985
Po - 210	0.005	0.003	5.000	Mar. 15, 1985
Po - 210	0.008	0.003	5.000	Mar. 15, 1985
Th - 230	0.01	0.01	2.000	
Th - 230	0.01	0.01	2.000	
Ra - 226	0.003	0.003	10.008	
Ra - 226	0.003	0.003	10.002	
Cs - 137	0.11	0.02	18.92	Mar. 26, 1985
Cs - 137	0.11	0.02	19.62	Mar. 26, 1985
U	0.03 ppm	0.02 ppm	2.87	
U	0.04 ppm	0.02 ppm	2.57	
U	0.03 ppm	0.02 ppm	2.74	
U	0.04 ppm	0.02 ppm	2.87	
U	0.04 ppm	0.02 ppm	2.57	
U	0.03 ppm	0.02 ppm	2.74	

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TABLE OF RESULTS

FISH BONE

Isotope	Activity (Bq/g)	Uncertainty (Bq/g, 95%C.L.)	Subsample Mass(g)	Date of Analysis
Pb - 210	L0.010	—	5.000	Mar. 19, 1985
Pb - 210	L0.010	—	5.000	Mar. 19, 1985
Po - 210	0.005	0.003	5.000	Mar. 15, 1985
Po - 210	0.007	0.003	5.000	Mar. 15, 1985
Th - 230	L0.01		0.25	
Th - 230	L0.01		0.25	
Ra - 226	0.003	0.003	5.000	
Ra - 226	0.003	0.003	2.000	
Cs - 137	0.030	0.005	53.60	Mar. 26, 1985
Cs - 137	0.032	0.005	53.25	Mar. 26, 1985
U	0.10 ppm	0.01 ppm	6.15	
U	0.09 ppm	0.01 ppm	5.96	
U	0.09 ppm	0.01 ppm	6.33	
U	0.09 ppm	0.01 ppm	6.15	
U	0.11 ppm	0.01 ppm	5.96	
U	0.09 ppm	0.01 ppm	6.33	

Note: L denotes a less than.

Radionuclide Methodology

The radionuclides analyzed as part of this contract include Pb-210, Po-210, Th-230, Ra-226, Cs-137 and uranium. The methods used for each radioisotope are outlined below. For those isotopes which were quantified by either gross alpha or gross beta counting techniques following radiochemical separations, the measurements were performed using a Canberra Model 2200 Alpha, Beta Analyzer. This instrument consists of an external proportional counter with an ultra-thin detector window. The counter is completely surrounded by four inches of virgin lead and utilizes ultrahigh purity P-10 counting gas (90% argon - 10% methane).

A. Pb-210:

Because of the weak beta emission from Pb-210, the determination of Pb-210 was carried out indirectly by measurement of its high energy beta-emitting daughter Bi-210.

A mixed carrier solution containing stable lead and bismuth was added to the 5.000 gm samples which were then digested to dryness with a mixture of nitric, hydrofluoric and perchloric acids. The bone samples were digested in two 2.500 gm portions which were subsequently recombined. If any residue remained, the digestion was repeated to yield a clear digest when taken up in 2M HCL. Lead and bismuth were extracted into chloroform with diethyldithiocarbamate and then back extracted into aqueous solution. The

bismuth concentration of this aqueous solution was then determined by atomic absorption spectrophotometry to determine the recovery of bismuth (typically 95-100%) through the extraction procedure. The bismuth was subsequently precipitated and purified as the oxychloride. The precipitate was filtered through a 0.45u membrane filter in a ring and disc filter assembly, air-dried briefly and then covered with aluminum foil. The aluminum foil serves to screen out weak beta emission from Pb-210 as well as alpha emission from Po-210 which is carried through the procedure.

Samples were counted for 100 minutes and compared to the activities of standard Pb-210 and Bi-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch. The standard Pb-210 solution, with which Bi-210 is certified to be in equilibrium, was purchased from Amersham Radiochemical Ltd. and was certified to have a total uncertainty of not greater than 2.3%.

Ref: Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 19-22.

B. Po-210:

Samples (5.000 gm) for polonium analysis were digested in Teflon beakers with a mixture of nitric, hydrofluoric and perchloric acids. The mixture was taken to perchloric acid fumes but not to dryness. The bone samples were digested in two 2.500 gm portions and subsequently recombined. The digested solutions were diluted, reduced with ascorbic acid then heated for two hours in contact with a silver disc. Under these conditions, polonium spontaneously deposits onto the silver disc which is subsequently counted for its alpha activity. Samples were counted for 100 minutes and

compared to the activity of standard Po-210 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch. The standard Po-210 solution was purchased from Amersham Chemicals and was certified to have a total uncertainty not exceeding 2.3%.

Ref. Canmet Report No. 78-22, G. Smithson, Ed. (1979) pp. 23-28.

C. Th-230:

Th-230 was determined by alpha spectroscopy using an Ortec Model 496 alpha spectrometer with two ruggedized silicon surface barrier detectors. The alpha spectrometer has a spectrum storage capability of 120 channels over a 3MeV range. The multichannel analyzer is controlled directly by a PDP 11/34 computer with 256K bytes of memory, four hard surface discs, a nine-track 1600 bpi tape drive and five different video or typewriter terminals.

Fish meal samples (10.000 gm) for Th-230 determination were spiked with beta-emitting Th-234 as a radiotracer, lanthanum chloride as a carrier and barium nitrate as a holdback carrier for radium. This mixture was then ashed at 550 Deg. C and the ash digested with nitric, hydrofluoric and perchloric acids until there was no undissolved residue. The fish bone samples (0.200-1.000 gms) were treated similarly except that no ashing step was performed. Thorium isotopes were then coprecipitated with lanthanum hydroxide, filtered, redissolved and coprecipitated with lanthanum fluoride. The thorium isotopes were then collected on 0.45u filters and alpha-counted for 100 minutes using the instrumentation described above. The samples were also beta-counted for Th-234 to

determine the overall yield of the separation. In addition one sample of each type was spiked with Th-230 to verify that counting efficiencies were not compromised by matrix self-absorption.

The activity of samples was compared with the activity of standard Th-230 solutions carried through the same procedure. Certified reference materials were also analyzed with each batch of samples. The standard Th-230 solution was obtained from Isotope Products Laboratories and had a total uncertainty not exceeding 5.95% on its calibration.

D. Ra-226:

Fish meal samples (10.000 gm) for Ra-226 were spiked with Ba-133 as a radiotracer and ashed at 550 Deg. C. The resulting ash was dissolved in a mixture of hot concentrated nitric, perchloric and hydrofluoric acids. Radium was precipitated from solution by coprecipitation with lead sulfate, redissolved and purified by coprecipitation with barium sulfate. The precipitate was collected, gamma counted for Ba-133 to measure the overall yield (90-100%) and then stored for a minimum of one week to allow for the ingrowth of daughters. At the end of this period, samples were counted for 100 minutes using gross alpha counting techniques and compared to the activity of standard Ra-226 solutions carried through the same procedure. The standard Ra-226 solutions were diluted from stock obtained from Amersham Radiochemicals. This solution had been previously calibrated against a U.S. National Bureau of Standards Ra-226 source and has a total uncertainty of not greater than $\pm 3.9\%$ in its activity.

Fish bone samples (5.000 gm) were treated similarly except that no ashing step was performed and the samples were digested in two 2.500 gm portions

which were subsequently recombined.

E. Cs-137:

Fish meal samples (approx. 20 gms) and fish bone samples (approx. 40 gms) were tightly packed in gamma planchets and counted for 50,000 secs. using a hyperpure 17% efficiency germanium gamma detector. The detector and sample are shielded by a lead cave of 15x15x20 inches. The walls of the cave are four inches thick and are lined with 0.02 inches of cadmium and 0.06 inches of copper to absorb low energy gamma rays. Data acquisition was carried out using an Ortec Model 7044 Multichannel Analyzer and the resulting spectrum was resolved using a PDP 11/34 computer system. Efficiency calibration was carried out using uranium ore (CANMET DL-1a) of known activity and identical counting geometry.

The accuracy of the calibration was confirmed by the analysis of the U.S. National Bureau of Standards River Sediment, SRM 4350B, a reference material containing numerous radioactive fission products including Cs-137.

F. Uranium:

Uranium analyses were performed by neutron activation analysis followed by delayed neutron counting. Fish meal samples (approx. 2.7 gms) and fish bone samples (approx. 6.0 gms) were irradiated for 60 seconds, cooled for 10 seconds, and counted for 60 seconds. Calibration was carried out using CANMET uranium standard DL-1a and vegetation standard CLV-1. The accuracy of the calibration was confirmed using U.S. National Bureau of Standards Tomato Leaves 1573, Coal 1535 and Pine Needles 1575. Six replicate

analyses were performed for each sample type due to the ease of the procedure.